

ADMINISTRATIVE RECORD

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE

APPROVED FOR USE IN LIBBY SUPERFUND SITE ONLY


1077384 - R8 SDMS

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SOP No. EPA-LIBBY-09

Title: STANDARD OPERATING PROCEDURE FOR TEM DATA REVIEW AND DATA
ENTRY VERIFICATION

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SYNOPSIS: This standard operating procedure provides a standardized method for review of raw TEM data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses for review and verification, b) review of the original laboratory TEM bench sheets, and c) verification of the transfer of results from the bench sheets into the Libby2 Database. This method is applicable for use only at the Libby Superfund Site.

APPROVALS:

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12/7/06

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12/7/06

Revision	Date	Reason for Revision
0	12/7/06	--

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for review of raw transmission electron microscopy (TEM) data and verification of entry of TEM results into the Libby2 Database. Steps included in this SOP are: a) selection of TEM analyses that will undergo a data consistency review and verification, b) performing a consistency review of the original laboratory TEM bench sheets to verify that TEM analysts working on the Libby project are performing analyses in accord with project-specific recording rules, and c) verifying the correct transfer of results from the bench sheets into the Libby2 Database.

2.0 PERSONNEL QUALIFICATIONS

Personnel performing data review and verification under this SOP must be skilled and/or trained in interpretation of raw data sheets and electronic data files in support of TEM analysis for the Libby Superfund Site. Personnel must be well-versed in TEM counting rules and Libby project-specific counting and recording rules in order to perform the required consistency reviews.

3.0 APPLICABILITY

A representative portion of TEM data, analyzed for the Libby Superfund Site, will be selected for review and verification to ensure consistency in data collection and data entry. The frequency of samples selected for review is discussed in subsequent sections.

4.0 SELECTION OF TEM RECORDS FOR REVIEW

The goals for selecting a representative subset of TEM results for review and verification are provided below. Selections should be made to ensure representation across several areas: 1) the fraction of total samples analyzed by TEM; 2) the types of programs (SAPs, QAPPs, etc.) carried out at the Site; 3) the laboratories performing TEM analysis.

Total Samples. Over the course of the Libby project (that begins with the date of this approved SOP), a minimum of ten percent (10%) of all TEM analyses should be selected for review and verification. Samples will be selected in a manner that ensures representation across the different types of programs and the laboratories performing the TEM analysis.

Types of Programs. If there are important differences in sampling and analysis protocols between sampling programs, data reviews and verifications will be stratified by program. At the request of EPA, the frequency of data review may be increased for specific programs of interest (i.e., investigative samples associated with ambient air monitoring, activity-based sampling, and cleanup efficacy evaluations). Of specific interest is ensuring reviews are stratified across programs that reflect differences in structure recording and/or counting rules.

Laboratories performing TEM analysis. Data reviews and verifications will be performed for each laboratory participating in TEM analysis in support of the Site sampling programs.

Specific details for selecting TEM records for review are outlined below.

1. On the 1st of each month, compile a list of all TEM ISO 10312 and all TEM AHERA/ASTM samples for which new results were uploaded into the Libby2 Database in the preceding month (e.g., on November 1st, specify a date range of Oct 1-31). Samples will be selected for review separately for TEM ISO 10312 and AHERA/ASTM.

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The Libby2 Database query will be based on the analysis upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date are not excluded. For example, consider the case where the TEM ISO 10312 analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The selection query performed on November 1, if limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

2. A minimum of 10% of all TEM ISO 10312 and TEM AHERA/ASTM analyses will be selected for review each month. To the extent practical, these will be first stratified by analyst, with the number of samples from each analyst being in proportion to the total number of samples analyzed by each analyst. If there are important differences between sampling programs (e.g., differences in counting and/or recording protocols), samples will also be stratified by program. In addition, samples will be stratified according to detect/non-detect, with approximately 50% of the samples selected being detects, and 50% being non-detects. The following table illustrates the selection process:

Analyst	Analyzed			Selected		
	Detect	ND	Total	Detect	ND	Total
1	14	112	126	11	6	17
2	20	421	441	16	22	38
3	2	4	6	2	1	3
4	0	8	8	0	1	1
Total	36	545	581	29	30	59

	Goal	Actual
Total	58	59
Detect	29	29
Non-detect	29	30

In this example, there are a total of 581 new TEM ISO 10312 analyses available for the month (36 detects + 545 non-detects), analyzed by four analysts. Thus, the total number of TEM ISO 10312 analyses to be selected for review is $10\% \cdot 581 = 58.1$ (rounded to 58). This total is to be split evenly between detects (29) and non-detects (29). The number of detects and non-detects selected per analysis is calculated by multiplying the target number (29) by the fraction of the total detects and non-detects evaluated by the analyst. For example, for Analyst 1:

$$\text{Number of detects} = 29 \cdot (14/36) = 11.3 \text{ (rounded to 11)}$$

$$\text{Number of non-detects} = 29 \cdot (112/545) = 5.9 \text{ (rounded to 6)}$$

If an analyst has analyzed at least one sample in a category (detect or non-detect), the minimum number of samples to be selected is one. For example, for Analyst 4, the number of detects analyzed is zero, so the number of detects selected is zero. For non-detects, the number to be selected (computed using the approach above) is:

$$\text{Number of non-detects} = 29 \cdot (8/545) = 0.4 \text{ (rounded to 0)}$$

In this case, the number selected is set to the minimum of 1.

As seen, this procedure will tend to select a higher proportion of detects (29 of 36 analyses, 81%) than non-detects (30 of 545 analyses, 6%). This approach is used

because it is considered likely that the incidence of errors may tend to be higher in samples with one or more detected structures than in samples with no detected structures.

3. Stratify the list of newly uploaded samples according to program (if applicable), analyst, and detection status (detect, non-detect), and select the appropriate number of samples for each category at random.
4. Based on the samples selected for review, create a list of all the unique analytical laboratory jobs which will be needed to review the selected analyses. Submit the list of analytical laboratory jobs to EPA's project file manager (Volpe).
5. Volpe will provide SRC with electronic copies (as Adobe Acrobat PDFs) of the requested analytical laboratory jobs via CD, an FTP site, or another electronic transfer mechanism.

5.0 CONSISTENCY REVIEW OF LABORATORY BENCH SHEETS

The purpose of the consistency review is to inspect data entered on the laboratory bench sheets in order to identify the occurrence of any data omissions, apparent inconsistencies, or potential errors in structure.

5.1 Consistency Review Procedure for TEM ISO 10312

1. For each TEM ISO 10312 analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.
2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with ISO 10312 counting rules (as modified in Libby Laboratory Modification LB-000016). The types of information that will be reviewed include:
 - The recorded structure types are consistent with the counting rules. Valid structure types include F, B, CC, CD, CF, CR, MC, MD, MF, and MR.
 - Disperse complex structures are broken down in accord with ISO 10312 counting rules and compact complex structures are not broken down. For example, a CD43 should provide 4 secondary structures, with 3 secondary structures greater than 5 um. In this example, the structure type for each of the recorded secondary structures should begin with the "C" prefix (e.g., CF, CB, CR).
 - The primary and total columns have been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.
 - If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.
 - All recorded fibers (F, CF, and MF) meet the 3:1 aspect ratio requirement.
 - The mineral class is populated for all structures.
 - Structure comments (e.g., < 3:1) are supported by recorded data.
 - The stored values in the Libby2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.2 Consistency Review Procedure for TEM AHERA/ASTM

1. For each TEM AHERA/ASTM analysis to be reviewed, locate the original hand-written laboratory bench sheet(s) within the appropriate laboratory job.
2. Review the original hand-written laboratory bench sheets to determine if the raw structure data are recorded in accord with AHERA/ASTM counting rules (as modified in Libby Laboratory Modification LB-000031). The types of information that will be reviewed include:
 - The recorded structure types are consistent with the counting rules. For AHERA/ASTM, valid structure types include F, B, M, and C.
 - The total column has been populated with non-zero numbers for all countable structures and a zero for all non-countable structures.
 - If recorded, all non-asbestos mineral (NAM) structures are identified as non-countable structures.
 - The recorded structures meet the counting rule requirements. For AHERA/ASTM, all recorded fibers and matrices meet the 5:1 aspect ratio requirement.
 - The recorded dimensions for matrices are the protrusion dimensions, not the matrix dimensions (provided sketches will be used to qualitatively assess dimensions).
 - The mineral class is populated for all structures.
 - Structure comments (e.g., < 5:1) are supported by recorded data.
 - The stored values in the Libby 2 Database for primary, total, structure type, length, width, and mineral class match the original bench sheet.

5.3 Corrective Action

The data reviewer will prepare a list of any apparent inconsistencies, omissions, or other suspected errors. This list will be provided to EPA and to the Libby laboratory coordinator (CDM), who will forward the list to the appropriate laboratories and analysts for review and response.

At the laboratory, the analyst that performed the analysis and the Quality Assurance (QA) personnel that signed off on the TEM electronic data deliverable (EDD) will review the issues identified and determine which of the issues identified are authentic errors that require correction. All errors will be corrected and a revised TEM EDD and/or hard copy bench sheet will be submitted to the Libby laboratory coordinator (CDM). Each laboratory will provide re-training for analysts and QA reviewers, as needed, to minimize the occurrence of errors at the level of the bench sheet and EDD.

6.0 VERIFICATION OF DATA TRANSFER FROM BENCH SHEET TO DATABASE

6.1 Verification Procedure

The purpose of verification is to ensure that the data from the bench sheet have been transferred into the Libby 2 Database without error or omission. The following steps will be performed as part of the data verification procedure.

1. Compare the analysis-specific information provided in the Libby2 Database to the original lab job documentation (e.g., internal laboratory chain of custody, preparation logs, etc.). [Note: Whenever possible, verification will be performed against hand-written notations, NOT internal laboratory summary tables prepared from hand-written notes. Every attempt

should be made to obtain the original hand-written notes. If laboratory summary tables are used instead of hand-written notes, this should be documented and specific rationale should be provided.] The following fields will be verified:

- Analysis Method (TEM-ISO10312, TEM-AHERA, ASTM)
- Analysis Date
- Lab Name
- Lab Job Number
- Lab Sample Number
- Preparation Method (Direct, Indirect, or Indirect with Ashing)
- Filter Status (Analyzed, Overloaded, Damaged, Missing, Cancelled)
- Primary Effective Filter Area (EFA, mm²)
- Secondary EFA (mm²) [For indirect preparations only]
- Grid Opening Area (Ago, mm²)^a
- F-factor [For indirect preparations only, direct prep F-factor = 1]
- Air Volume (L) or Sample Area (cm²)^b
- Analysis Comments

2. Verify the calculation of the F-factor for indirect preparations as follows:

F-factor = Fraction of primary filter used • Volume of resuspension fluid applied to secondary filter
/ Total resuspension volume

3. Verify the amphibole sensitivity recorded in the Libby2 Database as follows:

Air Sensitivity = EFA / (GOx • Ago • V • 1000 • F-factor)

Dust Sensitivity = EFA / (GOx • Ago • SA • F-factor)

where: EFA = Effective Filter Area (mm²)^c
GOx = Grid Openings Counted for Libby amphibole
Ago = Area of a Grid Opening (mm²)
V = Air Volume (L)
SA = Dust Sample Area (cm²)
F-factor = indirect preparation dilution factor

4. Count the total number of unique grid openings evaluated in the original hand-written laboratory bench sheets, and compare to the number in the field titled "AnalysisGOCounted" in the Libby2 Database. [Note: If more than one analysis has been performed for the same sample, determine if the grid openings recorded in the second analysis were inclusive or exclusive of the grid openings in the first analysis. This check helps identify cases where an updated or revised EDD is added to the database as a new file rather than replacing (overwriting) an old file, thereby resulting in the duplication of some data.]
5. Using the original hand-written laboratory bench sheets, count the total number of "countable" Libby amphibole (LA) structures across all grid openings evaluated, and compare this number with the "binned" LA values stored in the Libby2 Database.

^a If the grid opening area is not within the expected range (0.005 - 0.015 mm²), the value should be confirmed with the laboratory.

^b To account for potential rounding issues, if the reported analysis air volume or sample area different from the value reported for the sample but is within 0.5% this will be noted in the summary report, but the value will be considered to be correct.

^c For direct preparations this will be the primary EFA. For indirect preparations, this will be the secondary EFA.

- For ISO 10312 analyses, LA counts will be compared to Bin G for LA, which is equal to the total number of countable LA.
- For AHERA/ASTM, LA counts will be compared to the "S<5um" and "S>5um" bins for LA.

6.2 Corrective Action

For each sample where an issue has been identified, the data reviewer will obtain a hard copy of the laboratory bench sheet. Based on a review of the bench sheet, each issue will be classified as either a) an omission or data entry error at the level of the EDD, or b) an error at the level of the data upload from the EDD into the Libby2 Database.

The data reviewer will prepare a list of any noted discrepancies or omissions for each sample, along with the apparent type of error. This list will be provided to EPA and to the Libby laboratory coordinator (CDM) for review and response.

In cases of apparent data omission or error at the level of the EDD preparation, the laboratory coordinator will contact the laboratory and identify the apparent error(s). At the laboratory, the individual responsible for data entry from the bench sheet into the EDD and the QA personnel that signed off on the EDD will review the issue and make corrections to the EDD as needed. If corrections are made, a revised EDD will be submitted to EPA's database manager for re-entry into the Libby 2 Database. Re-training of data entry and QA review personnel may be implemented, as needed.

If the error is due to a database upload error, EPA's database manager (Volpe) will be contacted and notified of the issue. At Volpe, the TEM upload procedure will be reviewed to identify the source of the issue and modified to ensure that future TEM EDDs will be uploaded correctly. Depending on the nature of the issue, it may be necessary to identify other TEM analyses in the Libby 2 Database that would have been similarly impacted. Any potentially impacted TEM analyses should be removed from the Libby2 Database and re-uploaded after the upload procedure has been corrected.

7.0 REPORTING

The data reviewer will prepare a report which summarizes the results of the consistency review and data verification for the sample set and identifies areas for improvement. Attachment A provides an example of this report. As seen, this report includes a detailed summary of the consistency review and data verification findings, and includes a summary of the potential implications of the review and verification findings on the data quality and use of the TEM analyses in the Libby2 Database. This report will also provide copies of all electronic spreadsheets generated which track any identified discrepancies and the resolution status of each issue.

Based on the results of the review and verification, EPA may choose to modify (either increase or decrease) the frequency of TEM samples selected for review and verification and/or the selection/review/verification process.

8.0 REFERENCES

Asbestos Hazardous Emergency Response Act (AHERA). 1986. Title 20, Chapter 52, Sec. 4011. Public Law 99-519.

American Society for Testing and Materials (ASTM). 2003. Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for

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Asbestos Structure Number Concentrations. ASTM D 5755-03. American Society for Testing and Materials. October 2003.

International Organization for Standardization (ISO). 1995. Ambient Air – Determination of asbestos fibres – Direct-transfer transmission electron microscopy method. ISO 10312:1995(E).

ATTACHMENT A

**EXAMPLE OF TEM CONSISTENCY REVIEW
AND DATA TRANSFER VERIFICATION REPORT**

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

Date: _____ Prepared by: _____

Reporting Date Range: _____

SUMMARY OF FINDINGS AND DATA QUALITY IMPLICATIONS

Recommendations for future review and verification: _____

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

TEM-ISO 10312 SELECTION AND CONSISTENCY REVIEW RESULTS

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-ISO 10312 Analyses			Number of Analyses Selected for Review		
	Detect	Non-Detect	Total	Detect	Non-Detect	Total
1						
2						
3						
...						
Total						

	<u>Goal</u>	<u>Actual</u>
Selected Total	_____	_____
Selected Detects	_____	_____
Selected Non-Detects	_____	_____

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____ % of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

Number of analyses with recording issues identified: _____ (_____ % of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

- _____ Reported structure types are inconsistent with ISO guidance
- _____ Primary and/or total columns are not populated correctly
- _____ NAM structures are recorded and not identified as non-countable
- _____ Fibers recorded as countable do not meet 3:1 aspect ratio criteria
- _____ Mineral class designation is missing or inconsistent
- _____ Structure comments are inconsistent with recorded data
- _____ Structure attributes in the database do not match the bench sheet

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

TEM-AHERA/ASTM SELECTION AND CONSISTENCY REVIEW RESULTS

Summary of available analyses for date range specified –

Analyst, Lab	Number of TEM-AHERA/ASTM Analyses			Number of Analyses Selected for Review		
	Detect	Non-Detect	Total	Detect	Non-Detect	Total
1						
2						
3						
...						
Total						

	<u>Goal</u>	<u>Actual</u>
Selected Total	_____	_____
Selected Detects	_____	_____
Selected Non-Detects	_____	_____

Detailed summary of bench sheet consistency review –

Number of analyses reviewed: _____ (_____ % of total analyses selected)

If not all analyses could be reviewed, provide a brief explanation for why: _____

Number of analyses with recording issues identified: _____ (_____ % of total analyses reviewed)

Types of recording issues identified (indicate the number of analyses):

- _____ Reported structure types are inconsistent with AHERA/ASTM guidance
- _____ Total column is not populated correctly
- _____ NAM structures are recorded and not identified as non-countable
- _____ Fibers recorded as countable do not meet 5:1 aspect ratio criteria
- _____ Recorded dimensions for matrices are matrix dimensions not protrusion dimensions
- _____ Mineral class designation is missing or inconsistent
- _____ Structure comments are inconsistent with recorded data
- _____ Structure attributes in the database do not match the bench sheet

Do the recording issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

TEM CONSISTENCY REVIEW AND DATA TRANSFER VERIFICATION REPORT

DATA TRANSFER VERIFICATION RESULTS

Number of analyses verified¹: _____ (_____ % of total analyses selected)

Number of analyses with data transfer issues identified: _____ (_____ % of total analyses verified)

Types of data transfer issues identified:

- _____ Incorrect/missing information on analysis details (e.g., lab job number, analysis date, filter status)
- _____ F-factor calculation is incorrect or inputs are missing
- _____ Air volume or dust area reported by laboratory is inconsistent with field value
- _____ Number of grid openings counted is incorrect
- _____ Sensitivity calculation is incorrect or inputs are missing
- _____ Total number of countable LA structures is incorrect

Do the data transfer issues identified appear to be associated with a particular analyst or laboratory? Yes No

If yes, identify the analyst and/or laboratory: _____

Comments: _____

ISSUE RESOLUTION AND STATUS

¹ Only those analyses that have passed the bench sheet consistency review are included in the data transfer verification.